

Reply to Office action of April 22, 2003

REMARKS

This Amendment is respectfully submitted to place rejected claims of subject Application in condition for allowance. In particular, Claims 1, 6, 11 and 21 have been amended to more particularly point
5 out and distinctly claim the novel subject matter of the invention.

Applicants and their undersigned Attorney note with appreciation that Primary Examiner Griffin has withdrawn all previous rejections in view of amendments filed on February 11, 2003 with a declaration under 37 CFR 1.131 to overcome the Yen et al. (US
10 6,402,939) reference of record.

Claim 1 has been amended to further recite the step of treating at least a portion of the separated organic liquid by contact with at least one immiscible liquid comprising a solvent having a dielectric constant in a range from about 24 to about 80 suitable to selectively
15 extract oxidized sulfur-containing and/or nitrogen-containing organic compounds. Support for this amendment is found in the specification of subject application, for example at page 13, lines 13 to 24.

Claim 6 has been amended to further recite that the recovering of product from the treated organic liquid includes use of at least one
20 solid sorbent comprising silica, and at least a portion of the separated aqueous phase is recycled to the reaction mixture. Support for these amendment is found in the specification of subject application, for example at page 13, lines 10 to 12, and Claim 18.

Claim 11 has been amended to further recite a high-boiling
25 oxidation feedstock consisting essentially of a sulfur-rich, mono-aromatic-lean fraction boiling between about 200° C. and about 425° C. Support for these amendment is found in the specification of subject application, for example at page 12, lines 22 to 25, and Claim 9.

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Claim 21 has been amended to further recite the step of blending at least a portion of the low-boiling fraction with the product containing less sulfur and less nitrogen than the oxidation feedstock to obtain components containing less than about 50 parts per million of sulfur for refinery blending of a transportation fuel. Support for this amendment is found in the specification of subject application, for example at page 31, lines 25 to 28, and Claims 8 and 19.

103 Rejections

In outstanding Office Action, Claims 1 to 19, and 21 were rejected under 35 U.S.C. § 103(a) as being unpatentable over U.S. Patent No 6,217,748 in the name of Shigeto Hatanaka, Osamu Sadakane, and Hideshi Iki (Hatanaka et al.), in view of EP 0 482 841 A1 in the name of Frances Mary Collins, Andrew Richard Lucy, and David John Harry Smith (EP 0 482 841 A1), and U.S. Patent No 3,341,448 in the name of John Frederick Ford, Timothy Arrowsmith Rayne and Dennis George Adlington (Ford et al.). Applicants respectfully traverse this rejection.

The Hatanaka et al. reference of record describes a four-step hydrodesulfurization (HDS) of a sulfur-containing petroleum hydrocarbon diesel gas oil. The gas oil is catalytically hydrodesulfurized at 300° C. to about 500 ppm sulfur in a first HDS step. Step two is distillation at a cut point temperature in a range of 320 to 340° C. Step three is a second HDS at 320 to 380° C., but of only the heavy fraction of the distillation. The twice hydrodesulfurized heavy fraction (90 ppm sulfur) is mixed with the light fraction of the distillation (10 ppm sulfur) to a sulfur content of up to 0.01% (50 ppm sulfur, Hatanaka et al. Example 3).

Obvious disadvantages of the Hatanaka et al. process include the costs of the additional hydrogen and energy required for both the distillation and further hydrodesulfurization steps. While Hatanaka et al. state the hydrogen sulfides from the two HDS steps may be

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removed from the off-gas by an amine absorption arrangement, carbon oxides are also by-products of the HDS steps. Generation of the additional hydrogen also produces more carbon dioxide. These are especially critical at a time when most industries are striving to limit
5 the amount of emitted "green house" gas such as carbon dioxide.

By contrast Applicants' novel process includes a catalyzed, hydrogen peroxide, promoted oxidative desulfurization of a sulfur-rich, mono-aromatic-lean fraction without further production of carbon dioxide or hydrogen sulfides. An additional advantage of
10 Applicants' novel oxidative desulfurization process is that oxidation is selective to sulfur and nitrogen containing heteroaromatic compounds, i.e. does not saturate the non-sulfur, non-nitrogen multi-ring aromatic hydrocarbons.

The EP 0 482 841 A1 reference of record describes a process for
15 de-sulfurising hydrocarbon oils by catalytic oxidation and subsequent separation of oxidized sulfur from the treated hydrocarbon oil. The examples in EP 0 482 841 A1 report recovery of products that had retained sulfur levels of 0.27 to 0.085 percent by weight (2,700 ppm to 850 ppm)..

By contrast, examples of applicants' novel process demonstrate products having unexpectedly low levels of retained sulfur and nitrogen, typically less than one part per million sulfur and less than 5 ppm nitrogen. For example, attention of Examiner is invited to page 31, lines 26 to 28 of instant specification where applicants report
20 recovery of product that had retained levels of only 0.3 ppm sulfur and 2.5 ppm nitrogen. It is the position of applicants that the EP 0 482 841 A1 reference of record does not disclose or suggest these unexpectedly low levels of retained sulfur and nitrogen that are provided by applicants' novel process.

30 Rather than disclosing or suggesting the novel process of Applicants, the EP 0 482 841 A1 reference of record teaches a process

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which became obsolete after 1993. At most, the EP 0 482 841 A1 and Hatanaka et al references of record in combination establish a present need for an improved process for desulphurization of blending components for diesel fuel.

- 5 Applicants respectfully request Primary Examiner Griffin to withdraw rejections under 35 U.S.C. § 103 (a) which rely upon the Hatanaka et al. reference in combination with EP 0 482 841 A1 .

10 The Ford et al. reference of record is directed to a desulfurization of a petroleum residue containing a least a portion of material boiling above 250° C. and having at least 1 percent by weight of sulfur as sulfur-containing compounds by two successive stages. Their first stage is a combination of an oxidative step and a thermal desulphurization step, and the other stage is a hydrocatalytic desulphurization of the total effluent from the first stage.

- 15 After contacting the feedstock with molecular oxygen or air as the oxidizing agent, preferably with solid metal promoter at 130 to 180° C. for 2 to 20 hours, any oxidized sulfur compounds are said to be eliminated as gaseous decomposition products from the reaction mixture by thermal decomposition at temperatures of up to 400° C.
20 within 1/2 to 5 hours. Clearly the gaseous decomposition products of the Ford et al. process are a potential source of atmospheric pollution.

- At best Ford et al. report their first stage removed only 30 percent of the sulfur based on untreated feedstock. While their hydrocatalytic desulphurization stage further removed 30 percent,
25 their process removed only 60 percent of the sulfur based on untreated feedstock. Feedstock of at least 1 percent by weight of sulfur treated by the first of Ford et al. is expected to contain at least 0.7 percent of sulfur (7,000 ppm), and the overall two stage process of Ford et al. is expected to contain at least 0.4 percent of sulfur (4,000
30 ppm).

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While in the past a typical specifications for Diesel fuel was a maximum of 0.5 percent by weight, by 1993 legislation in Europe and United States limited sulfur in Diesel fuel to 0.3 weight percent. By 1996 in Europe and United States, and 1997 in Japan, maximum sulfur
5 in Diesel fuel was reduced to no more than 0.05 weight percent. This world-wide trend must be expected to continue to even lower levels for sulfur.

Rather than disclosing or suggesting the novel process of Applicants, the Ford et al. reference of record teaches a process which
10 became obsolete after 1993.

Available evidence strongly suggests that ultra-low sulfur fuel is a significant technology enabler for catalytic treatment of diesel exhaust to control emissions. Fuel sulfur levels of below 15 ppm, likely, are required to achieve particulate levels below 0.01 g/bhp-hr.
15 Such levels would be very compatible with catalyst combinations for exhaust treatment now emerging, which have shown capability to achieve NOx emissions around 0.5 g/bhp-hr. Furthermore, NOx trap systems are extremely sensitive to fuel sulfur and available evidence suggests that they need would need sulfur levels below 10 ppm to
20 remain active. Therefore, a critical element of Applicants' novel process is reduction of nitrogen.

By contrast, working Examples of Applicants' novel process demonstrated that their process removed more than 95 percent of the of the sulfur and nitrogen (See Example 3 where 99.3 percent sulfur
25 was removed, i.e., from 48 ppm sulfur and 49 ppm nitrogen to 0.3 ppm sulfur and 2.5 ppm nitrogen, Example 4 where 97.5 percent sulfur was removed, i.e., from 24 ppm sulfur and 16 ppm nitrogen to 0.3 ppm sulfur and 2.5 ppm nitrogen, and Example 5 where 99.4 percent sulfur was removed, i.e., from 70 ppm sulfur to 0.4 ppm
30 sulfur).

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Applicants respectfully request Primary Examiner Griffin to withdraw all rejections under 35 U.S.C. § 103 (a) which rely upon the Hatanaka et al. reference in combination with the Ford et al.

5 Claim 20 was rejected under 35 U.S.C. § 103(a) as being unpatentable over U.S. Patent No 6,217,748 (Hatanaka et al.), in view of EP 0 482 841 A1 and U.S. Patent No 3,341,448 (Ford et al.) as applied to Claim 17 above, and further in view of U.S. Patent No. 3,909,395 in the name of Edward Andrew Takacs (Takacs). Applicants respectfully traverse this rejection.

10 The Takacs reference of record, describes a four step process for upgrading a malodorous sulfur-contaminated mixture, for example, crude sulfate turpentine, crude sulfated pine oil, sulfur contaminated anethole or gasoline, for example (i) oxidation, (ii) washing with water, (iii) washing with aqueous sodium carbonate solution, and (iv)
15 subsequent steam distillation of a starting-pine oil containing 5,200 ppm sulfur to obtain a product containing 319 ppm of sulfur (Column 3, lines 1 to 16).

Examiner avers that the Takas reference suggests that by washing a treated liquid (pine oil not a mixture derived from
20 petroleum feedstock by fractionating the petroleum feedstock according to instant novel claims) because the washed liquid will have a reduced amount of sulfur components. However, in the Takacs treatment of foul smelling, sulfur-contaminated gasoline to obtain a "sweet" smelling gasoline, the step of washing with aqueous sodium
25 carbonate solution, step (iii), was omitted. Clearly Takas teaches that a step of washing with aqueous sodium carbonate solution is **not** useful to treat a petroleum derived mixture such as gasoline.

Applicants respectfully request Primary Examiner Griffin to withdraw all rejections under 35 U.S.C. § 103 (a) which rely upon
30 Takacs reference either taken alone or in combination with Hatanaka

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et al. in combination with the EP 0 482 841 A1 and Ford et al. references of record.

Applicants agree with Examiner that the references of record but not applied do not disclose or suggest Applicant's novel subject matter which includes, for example,

- (i) hydrotreating a petroleum distillate consisting essentially of material boiling between about 50° C. and about 425° C. by a process which includes reacting the petroleum distillate with a source of hydrogen at hydrogenation conditions in the presence of a hydrogenation catalyst to assist by hydrogenation removal of sulfur and/or nitrogen from the hydrotreated petroleum distillate;
- (ii) fractionating the hydrotreated petroleum distillate by distillation to provide at least one low-boiling blending component consisting of a sulfur-lean, mono-aromatic-rich fraction, and a high-boiling oxidation feedstock consisting essentially of a sulfur-rich, mono-aromatic-lean fraction boiling between about 200° C. and about 425° C.;
- (iii) contacting the high-boiling oxidation feedstock with a soluble quaternary ammonium salt containing halogen, sulfate, or bisulfate anion, and an immiscible aqueous phase comprising a source of hydrogen peroxide, and at least one member of the group consisting of phosphomolybdic acid and phosphotungstic acid, in a liquid reaction mixture under conditions suitable for reaction of one or more of the sulfur-containing and/or nitrogen-containing organic compounds;
- (iv) separating from the reaction mixture an essentially organic liquid and at least a portion of the immiscible aqueous phase; and
- (v) treating at least a portion of the recovered organic liquid with a solid sorbent, an ion exchange resin, and/or a suitable immiscible liquid containing a solvent or a soluble basic chemical compound, to obtain a product containing less sulfur and less nitrogen than the oxidation feedstock.

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Applicants urge that Claims 1 to 21, all claims now presented, are in condition for allowance. Applicant respectfully requests Examiner to pass subject application for allowance.

5 Do not hesitate to contact Frederick S. Jerome whose telephone number is (630) 832-7974 (FAX (630) 832-7976) if additional assistance is needed regarding this paper or earlier papers for Applicants.

Applicants and their undersigned Attorney appreciate Examiner's attention and further consideration of this matter.

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Respectfully submitted,



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